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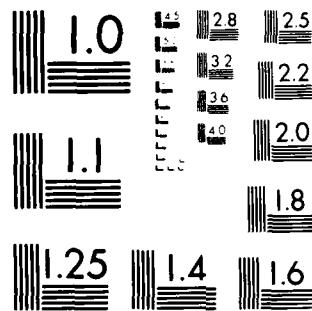
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## PREPARATION OF SOLID ELECTROLYTES

BY LARRY E. DEVRIES  
RESEARCH AND TECHNOLOGY DEPARTMENT

FEBRUARY 1983

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) <table> <tr><td>Solid Electrolyte</td><td>Copper(I) Iodide</td></tr> <tr><td>Silver Iodide</td><td>Solid State Preparation</td></tr> <tr><td>Quaternary Ammonium Iodides</td><td>Melt Preparation</td></tr> <tr><td>Trimethylsulfonium Iodide</td><td>Refluxing Solvent Preparation</td></tr> <tr><td>Thallium(I) Iodide</td><td>Paste Preparation</td></tr> </table>			Solid Electrolyte	Copper(I) Iodide	Silver Iodide	Solid State Preparation	Quaternary Ammonium Iodides	Melt Preparation	Trimethylsulfonium Iodide	Refluxing Solvent Preparation	Thallium(I) Iodide	Paste Preparation
Solid Electrolyte	Copper(I) Iodide											
Silver Iodide	Solid State Preparation											
Quaternary Ammonium Iodides	Melt Preparation											
Trimethylsulfonium Iodide	Refluxing Solvent Preparation											
Thallium(I) Iodide	Paste Preparation											
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>Several methods have been studied for preparing solid electrolytes involving silver iodide, thallium(I) iodide, and copper(I) iodide with quaternary ammonium iodides, ternary-sulfonium iodides, and pseudohalides. A range of compositions of silver iodide and trimethylsulfonium iodide gives electrolytes with specific conductivities close to 0.04 S/cm.</p>												

FOREWORD

Different methods were studied for the preparation of solid electrolytes. Silver iodide, thallium(I) iodide, and copper(I) iodide were used with quaternary ammonium iodides, a ternary sulfonium iodide, and pseudohalides. A solid state reaction method appears to be the best method of preparation. Using it, solid electrolytes containing silver iodide and trimethylsulfonium iodide over a range of compositions had specific conductivities near 0.04 S/cm. This work was funded by the Naval Air Systems Command under AIRTASK A05-532-005/216-1/W1120-01.

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## INTRODUCTION

The Electrochemistry Branch (Code R33) of the Naval Surface Weapons Center (White Oak) began testing solid electrolyte batteries in 1968 when batteries became available containing electrolytes with high ionic conductivities. Battery cells normally contain liquid electrolyte solutions to furnish ionic conductivity between the anode and cathode. Even "dry cells" contain an electrolyte solution which is absorbed in the separator, porous cathode, and in the anode when it is porous. However, liquid electrolytes often promote corrosion in battery cells especially with chemically active anodes. Solid electrolytes do not promote corrosion, but until the 1960's they had very low ionic conductivities which prevented their use in batteries. In 1972 a solid electrolyte battery with cells containing a silver (Ag) metal anode, an electrolyte made of silver iodide and rubidium iodide ( $\text{Ag}_4\text{RbI}_5$ ), and a cathode containing iodine complexed by tetramethylammonium iodide ( $(\text{CH}_3)_4\text{NI}_9$ ) was tested. The electrochemical reaction is  $2\text{Ag} + \text{I}_2 \rightarrow 2\text{AgI}$  for which the calculated open circuit voltage (OCV) is 0.686V. The measured OCV is 0.66V indicating that the iodine is complexed. Iodine diffusion is reduced by adding silver to the electrolyte as a getter. The test results at ambient conditions show that a 10 year shelf-life has been achieved (reference 1). However, this system has problems which could be eliminated by changing either the electrolyte or the electrolyte and anode. Under certain conditions the electrolyte is sensitive to water degredation because of the presence of rubidium iodide. Under other conditions it is light sensitive because of the silver iodide.

The cell OCV, 0.66V, is low and the cell cost high because silver is used. Silver compounds generally have small Gibbs energies of formation which means that silver used as an anode gives a low voltage. Other metals substituted for silver as the anode would give higher voltages. However, the (tetra)silver rubidium (pentakis)iodide ( $\text{Ag}_4\text{RbI}_5$ ) electrolyte decomposes at 0.67V. To avoid decomposition and maintain good ionic conductivity, the electrolyte must also be changed to contain metal ions of the metal substituted for silver. Substituting copper for silver as the anode would not raise the voltage too much although a copper containing electrolyte would be cheaper and more stable than the silver containing electrolyte now being used. The OCV would be about 0.69V with the cathode now used. However, substituting thallium for the silver anode would give an OCV of about 1.27V.

The water sensitivity of the electrolyte could be eliminated by substituting appropriate organic iodides for rubidium iodide. Ammonium iodide or other alkali metal iodides such as potassium can not be substituted for rubidium iodide since they are subject to the same water degredation as rubidium iodide. The substitution of an organic iodide for rubidium iodide might also stabilize

the silver iodide in the electrolyte against light sensitivity. The light sensitivity would be eliminated if an iodide such as thallium(I) iodide or copper(I) iodide were substituted for silver iodide. Substituting an organic iodide for rubidium iodide would lower the cost of materials in the electrolyte as would substituting either thallium(I) iodide or copper(I) iodide for silver iodide.

Because of the reasons given above, it was decided to investigate new solid electrolytes. A search was made for a more stable silver containing electrolyte using organic iodides in place of rubidium iodide. A search was also made for a cheaper and more stable electrolyte using primarily organic iodides and iodides such as copper(I) iodide and thallium(I) iodide in place of silver iodide. It might be possible to use better anode materials with such solid electrolytes, and if necessary perhaps other cathode materials not containing iodine could be substituted for the zero oxidation state-iodine containing cathode. Mercury (II) iodide used as a cathode would give a voltage of about 0.77V with a thallium anode. This would eliminate problems associated with the iodine cathode.

#### ORGANIC IODIDE-SILVER IODIDE SOLID ELECTROLYTES

Conductive solid electrolytes containing silver iodide and either potassium iodide, rubidium iodide, or ammonium iodide have been prepared (reference 2). Thus, it was logical to consider making conductive solid electrolytes containing silver iodide and various organic iodides such as quaternary ammonium iodides. Solid electrolytes containing such organic iodides as tetramethylammonium iodide or diethyldimethylammonium iodide have been reported to have conductivities as high as 0.04 to 0.06 S/cm (references 3 and 4). The references indicate that in the range of mole ratios of silver iodide to organic iodide of 4 to 1 up to 8 to 1 the conductivities vary little and generally go through a maximum. Using tetramethylammonium iodide, the conductivity of the corresponding electrolyte varies from about 0.01 to 0.04 S/cm. The maximum occurs at a ratio of about 5.7 moles of silver iodide to one mole of tetramethylammonium iodide.

The conductivities of electrolytes with quaternary ammonium iodides substituted for alkali metal iodides are lower than those of the alkali metal iodides or ammonium iodide. For the electrolytes with  $K^+$ ,  $Rb^+$ , or  $NH_4^+$  the conductivities are about 0.2 S/cm although a range of values has been reported (references 2, 5, and 6). However, the  $Ag_4RbI_5$  electrolyte with a conductivity as low as 0.1 S/cm is used in solid electrolyte batteries. Thus, an electrolyte with an organic iodide which had a conductivity of 0.1 to 0.15 S/cm would suffice.

## METHODS OF PREPARATION

Experiments were first carried out to gain experience with the preparation of silver iodide and organic iodide containing electrolytes. Several methods have been reported for preparing electrolytes containing organic iodides (references 3 and 4). The simplest method, the melt preparation method, involves heating the two iodides together until a liquid is formed. The liquid is stirred and then quenched. In another method of preparation, the solid state reaction method, the two powdered iodides are mixed together and pressed into a pellet. The pellet is placed in an oven and heated to a temperature below the melting point. After a few hours the pellet is pulverized. The powder is stirred, repelletized, and put back in the oven for a few hours. This process may be repeated several times until a conductive material is formed.

Two other methods of preparation involve the use of a liquid solvent. In one case, the refluxing solvent method, the iodides react in an excess of solvent kept at the boiling temperature of the solvent until the electrolyte precipitates out. In the other case, the paste preparation method, a paste is made of the iodides and a small amount of solvent. The solvent is evaporated from the iodides; and the solid is pulverized, pressed into a pellet, and put in an oven set at a temperature below the melting point. After several hours, the pellet is pulverized, repelletized, and put back in the oven until the reaction is complete. This method of preparation is a combination of the refluxing solvent method and the solid state reaction method.

## EXPERIMENTAL RESULTS AND OBSERVATIONS

Tables 1 and 3 contain the results of various attempts to prepare solid electrolytes. The experiment numbers serve only as a means of identification. In experiments numbered 1 through 4, the melt preparation method was used to try to prepare electrolytes containing silver iodide and quaternary ammonium iodides. The results were not as good as those reported in the literature, but the experience gained helped in later preparation attempts.

Literature reports on solid electrolytes made with silver iodide and either alkali metal iodides or quaternary ammonium iodides (references 3 and 4) indicated that ternary sulfonium iodides would probably make acceptable substitutes for rubidium iodide with a good possibility of forming solid electrolytes with a conductivity of 0.1 S/cm or higher. The solid state reaction method was used to prepare electrolytes containing trimethylsulfonium iodide and silver iodide. The ratios of moles of silver iodide to trimethylsulfonium iodide near 5.7 to 1 give conductivities of about 0.04 S/cm as indicated by the data for Experiment 5 in Table 1 and the data in Table 2. This is a new electrolyte which apparently has never been prepared before, and which seems to stop the sensitivity of silver iodide to light. Altering the structure of this ternary sulfonium iodide might give a more conductive material. Ethyldimethylsulfonium iodide would be a good ternary sulfonium iodide to try.

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TABLE 1. SILVER IODIDE, COPPER(I) IODIDE EXPERIMENTAL DATA\*

Components	Experiment Number	Mole Ratio	Pellet Thickness mm	AC Resistance	Conductivity S/cm	Method of Preparation	Temperature K	Comments
<b>AgI**</b>								
BTM	1	4/1	> 1 x 10 <sup>6</sup>	4.9	2.1 x 10 <sup>-3</sup>	M	413	TTL
BTM	2	6/1	1.298	72	2.6 x 10 <sup>-3</sup> ***	M	453	
BTM	3	8/1	2.332	20	5.3 x 10 <sup>-3</sup> †	M	458	
TMA	4	7/1	1.339	6.2	0.04	S	>473	
TMS	5	5.7/1	3.114	27	0.011	M	413-423	
RbI, AgCN	6	3/1/1	3.810	17	8.6 x 10 <sup>-3</sup>	M	About 548	PD
RbI, CuCN	7	3/1/1	1.854	3.4 x 10 <sup>4</sup>	M	About 573	PD	
RbI, AgSCN	8	3/1/1	1.557	4.4 x 10 <sup>3</sup>	M	523	PD	
KSCN	9	4/1	0.940	1.6 x 10 <sup>4</sup>	M	About 458	PD	
KSCN	10	2/1	0.889	1.6 x 10 <sup>4</sup>	M	About 458	PD	
<b>CuI**</b>								
BTM	16	6/1	1.740	1.2 x 10 <sup>3</sup>	M	About 473		
PTM	17	6/1	3.327	850	M	458		
TEA	18	6/1	2.416	3.5 x 10 <sup>5</sup>	M	573		
TMA	19	6/1	0.952	90	8.4 x 10 <sup>-4</sup>	M	548	
TMA	27	8/1	> 1 x 10 <sup>6</sup>	R††				
TMA	28	6/1	2.520	200	1 x 10 <sup>-3</sup>	S	413-423	NR
<b>8</b>								

\* Abbreviations: BTM = Benzyltrimethylammonium Iodide, M = Melt Preparation, NR = No Reaction, PD = Possibly Decomposition, PTM = Phenyltrimethylammonium Iodide, R = Refluxing Solvent, S = Solid State Reaction, TEA = Tetraethylammonium Iodide, TMA = Tetramethylammonium Iodide, TMS = Trimethylsulfonium Iodide, and TTL = Temperature Too Low.

\*\* Major component listed first in mole ratio.

\*\*\* The value in reference 2 for this composition is 0.01 S/cm.

† The value in reference 3 is 0.034 S/cm.

†† The solvent was 1-Hexanol, and the refluxing time was 3 hr.

TABLE 2. CONDUCTIVITY VERSUS COMPOSITION OF SILVER  
IODIDE-TRIMETHYLSULFONIUM IODIDE SOLID ELECTROLYTE

WEIGHT PERCENT SILVER IODIDE	MOLE RATIO SILVER IODIDE/TRIMETHYLSULFONIUM IODIDE	CONDUCTIVITY S/cm
82.14	4/1	0.010
85.18	5/1	0.011
86.35	5.5/1	0.036
86.77	5.7/1	0.036
86.83	5.73/1	0.040
86.87	5.75/1	0.035
87.34	6/1	0.026
88.95	7/1	0.022

TABLE 3. THALLIUM(I) IODIDE EXPERIMENTAL DATA\*

<u>Component</u>	<u>Experiment Number</u>	<u>Mole Ratio</u>	<u>AC Resistance</u>	<u>Method of Preparation</u>	<u>Temperature K</u>	<u>Solvent</u>	<u>Time hr</u>	<u>Comments</u>
<b>TlI**</b>								
BTM	11	4/1	>1x10 <sup>6</sup>	M	413			TTL
BTM	12	4/1	>1x10 <sup>6</sup>	M	613			D
BTM	13	2/1	>1x10 <sup>6</sup>	M	483			IPS
BTM	20	4/1	>1x10 <sup>6</sup>	R				
BTM	21	4/1	>1x10 <sup>6</sup>	R		1 - Hexanol	1	NR
TEA	14	4/1	>1x10 <sup>6</sup>	M	698	Acetone	1	NR
TEA	22	4/1	>1x10 <sup>6</sup>	R				TPS, D
TEA	23	4/1	>1x10 <sup>6</sup>	R		Benzene	3	NR
TEA	24	4/1	>1x10 <sup>6</sup>	R		Methanol	1	NR
TEA	25	4/1	>1x10 <sup>6</sup>	R		Water	3	NR
TPA	15	4/1	>1x10 <sup>6</sup>	M	583	Pyridine	1.5	NR
TPA	26	4/1	>1x10 <sup>6</sup>	R		IPS		
						1-Propanol	1.5	NR

\* Abbreviations: BTM = Benzyltrimethylammonium Iodide, D = Decomposition, IPS = Incomplete Phase Separation, M = Melt Preparation, NR = No Reaction, R = Refluxing Solvent, TEA = Tetraethylammonium Iodide, TPA = Tetrapropylammonium Iodide, TTL = Temperature Too Low.

\*\* Major component listed first in mole ratio.

Supposedly the addition of a pseudohalogen to the silver-rubidium iodide system gives a conductive material with different properties from the silver-rubidium system alone. A few preparations using cyanides and thiocyanates were attempted. The results of these experiments are listed in Table 1, Experiments 6 through 8. The melt preparation method was used in all cases with nitrogen passing over the melt. Although in each case the temperature was kept below the reported decomposition temperature for the pseudohalogen, there may have been decomposition in the melts. In the case of the cyanides cyanogen gas could be formed leaving metal in the melt. This would lower the resistance of the product and raise its conductivity. For thiocyanates a solid polymer of thiocyanogen is possibly formed. This would raise the resistance of the product. However, it may be that thiocyanates do not form conductive materials with silver iodide while cyanides form conductive materials. These experiments need to be repeated. It was found that the nitrogen flow rate was not high enough to prevent oxygen from getting into the melts. This would aid in the decomposition of some of the melts. Also it was discovered that the rubidium iodide contained a reducing agent which in aqueous solution at least, reduced silver ions to silver metal.

As seen in Table 1, potassium thiocyanate was substituted for rubidium iodide in two experiments. Experiment 10 had twice as much potassium thiocyanate as Experiment 9. This was to give a large volume of melt. Neither experiment gave a conductive product, however.

#### SOLID ELECTROLYTES WITHOUT SILVER

In trying to form new electrolytes not containing silver iodide, thallium(I) iodide and copper(I) iodide have been the main materials studied. This is because both of these materials have several properties similar to those of silver iodide. So far all attempts to use these salts in the melt preparation method for making electrolytes have failed. Experiments 11 through 15 in Table 3 give the data for the experiments with thallium(I) iodide. Although thallium(I) iodide has a lower melting point than silver iodide, it appears that many binary mixtures of thallium(I) iodide with other iodides have higher temperatures for melt formation than the corresponding mixtures with silver iodide. It was found that generally there was sublimation, distillation, and decomposition of the organic iodides when attempts were made to heat the mixtures to high enough temperatures to form a quantity of melt. Lower temperatures which gave very little liquid did not yield conductive materials. It was noted, however, that where a small amount of liquid phase was present there had not been a good separation of phases. Solid had been trapped in the liquid phase before it was quenched. It would be a good idea to attempt better phase separations and determine the resistances of the purer phases.

Experiments 16 through 19 in Table 1 gave data for melt preparation trials using copper(I) iodide in place of silver iodide. The results were similar to those observed for thallium(I) iodide. However, the material with tetramethylammonium iodide, Experiment 19, did give a conductivity of  $8.4 \times$

$10^{-4}$  S/cm. This method of preparation should be repeated using nitrogen over the melt. The solid state preparation, Experiment 28, indicated that maybe a conductive material could be made with copper(I) iodide and tetramethylammonium iodide.

Experiments 20 through 26 in Table 3 and 27 in Table 1 gave data for the refluxing solvent method for the preparation of electrolytes. There have been no reactions apparently between either thallium(I) iodide or copper(I) iodide and the organic iodides. In all cases it appeared that the organic iodide remained unreacted in the solvent, and the thallium(I) iodide or copper(I) iodide remained unreacted in the solid phase. There may have been a small amount of conductive material formed in one of the phases, but the bulk of the material from either phase was not conductive. The bulk of unreacted material would mask the conductivity of a small amount of conductive material. A mixed system of solvents or individual solvents not yet tried might permit a conductive material to form and precipitate out. Thallium(I) iodide did dissolve somewhat in acetone and 1-hexanol. It was not ionized, however, since these solvents formed yellow solutions. Thallium(I) iodide probably needs to be ionized to react and precipitate a conductive material.

At this time the paste preparation method has not been used. It is similar to the solid state reaction method. The solid state reaction method has only been used to a very limited extent. Experiment 28 in Table 1 indicated that there is a possibility of using the solid state reaction method to form a conductive material of copper(I) iodide and tetramethylammonium iodide. The conductivity was 0.001 S/cm. More experiments are needed to see if a much higher conductivity can be obtained.

#### SUMMARY

The aims of the experiments described in this report have been to find a substitute for rubidium iodide in the silver-rubidium iodide electrolyte and to make a completely new electrolyte containing neither silver nor rubidium. Three methods of preparation of solid electrolytes were evaluated. The melt preparation method was not successful in several experiments. The high temperatures needed promoted decomposition in some cases. In other cases incomplete phase separation occurred. The refluxing solvent method of preparation was not successful. The solvents tried did not dissolve both reactants in most cases, and no reaction took place. The solid state reaction method is the most promising method. The temperature can be kept low enough to avoid decomposition. Using the solid state method of preparation, the silver iodide-trimethylsulfonium iodide system gave an electrolyte with a specific conductance of 0.04 S/cm. Because of funding limitations, only a very short time was spent studying thallium(I) or copper(I) iodides as substitutes for silver iodide. The solid state method of preparation was used only with copper(I) iodide. Used with the copper(I) iodide-tetramethylammonium iodide system, it gave an electrolyte with a specific conductivity of 0.001 S/cm.

FUTURE EXPERIMENTS

The use of the solid state method of preparation involving lower temperatures than the melt preparation method should be used with thallium(I) and copper(I) iodides. Using this method of preparation with silver iodide and other organic iodides might produce a solid electrolyte more useful than the solid electrolyte in the batteries now being tested. It appears that ternary sulfonium iodides have a possibility of producing an electrolyte with a specific conductivity of 0.1 S/cm, and would increase the resistance of silver iodide to light induced decomposition.

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